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MICRO-TENSILE TESTING AND 3D-EBSD CHARACTERIZATION OF PURE NICKEL MULTI-CRYSTALS (Preprint)

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14. ABSTRACT
A dual beam focused ion beam-scanning electron microscope (DB FIB-SEM) outfitted with an electron backscatter diffraction (EBSD) system was applied to characterize the internal microstructure and local lattice rotations within multi-crystal micro-scale test samples. The methodology outlined in this paper provides a high-fidelity 3D characterization of the internal grain structure of mechanical test samples in conjunction with knowledge of the external boundary conditions and measurement of the resultant stress-strain behavior, as well as characterization of the internal lattice rotations that have developed.

15. SUBJECT TERMS
micro-tension, CP-FEM, 3D-EBSD

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ABSTRACT: A dual beam focused ion beam-scanning electron microscope (DB FIB-SEM) outfitted with an electron backscatter diffraction (EBSD) system was applied to characterize the internal microstructure and local lattice rotations within multi-crystal micro-scale test samples. The methodology outlined in this paper provides a high-fidelity 3D characterization of the internal grain structure of mechanical test samples in conjunction with knowledge of the external boundary conditions and measurement of the resultant stress-strain behavior, as well as characterization of the internal lattice rotations that have developed.

INTRODUCTION: The majority of structural components are fabricated from polycrystalline materials, and as such there exists a demand for modeling and simulation tools that can accurately predict the plastic deformation response of polycrystalline ensembles. One example of a simulation approach to address this need is the crystal plasticity finite element method (CP-FEM) combined with meshes that explicitly represent the morphology and local crystallographic orientations of polycrystalline microstructures. Experimental validation of such methods is critical to their further development. Yet, due to experimental and computational challenges, these studies have typically been limited to simulations of explicit microstructures without experimental comparison (Lewis et al. [2008]), experimental comparison to simulations of statistically informed microstructures (St-Pierre et al. [2008]), and experimental comparison to simulations with explicit representations of simplified structures (e.g., columnar grains where the sub-surface microstructure is assumed, or 3D measurements of only the near-surface grains) (Kalidindi et al. [2004]; Musienko et al. [2007]; Zhao et al. [2008]). In the present work, we demonstrate a methodology for generating high-fidelity micro-tension datasets with explicit microstructure representation, which can be coupled to simulations for validation and further model development.

PROCEDURE, RESULTS AND DISCUSSION: The material selected for examination was a 99.0% purity annealed Ni foil, purchased commercially with an initial thickness of 50 μm. Micro-tensile samples were fabricated from the foil by implementing a stencil mask technique. This technique involves using standard microelectronics processing methods to produce high aspect-ratio patterned templates, i.e. stencil masks, from a Si wafer. Once fabricated, the stencil masks are placed on top of a thin metallic substrate and the mask-and-substrate are co-sputtered using a broad ion beam milling system. This ultimately transfers the 2D pattern of the stencil mask onto the metallic substrate. The final sample geometry was defined using FIB milling, through an automated process that serially milled the perimeter surface of each sample while maintaining a back-tilt of 1° to ensure orthogonal sidewalls. The final sample geometry can be seen in Fig. 1.

Three samples with gage volumes containing approximately 200 grains and nominal gage dimensions of 21 x 38 x 80 μm were tested to different strain levels (~ 1.0, 2.5, and 12.5 % axial engineering strain) through in-situ SEM micro-tension experiments using a custom mechanical testing device. Despite the limited number of grains within the gage volume and expected variation in local grain configurations among the three samples, there is good agreement among the engineering stress-strain curves, as shown in Fig. 2. Surface strains were calculated using DIC analysis to track the motion of FIB-milled fiducial markers. The surface strain distribution was found to be heterogeneous, where some regions remain nearly undeformed while others contain strains which are more than double the average value.

The 3D microstructure and internal lattice rotations of the deformed samples were characterized following mechanical testing by destructive 3D-EBSD serial sectioning using custom automation software and an FEI Nova 600 DB FIB-SEM equipped with a TSL Hikari high speed EBSD detector. The cross-sections were milled with a section thickness of 250 nm. Crystallographic orientation information was captured for each section by collecting EBSD maps using a pixel size of 250 nm. The grain structure was subsequently reconstructed using DREAM 3-D software (http://dream3d.bluequartz.net/), an example of which is shown in Fig. 3. The reconstructed microstructure in low strain samples was subsequently meshed and used as the input for FEM simulations.

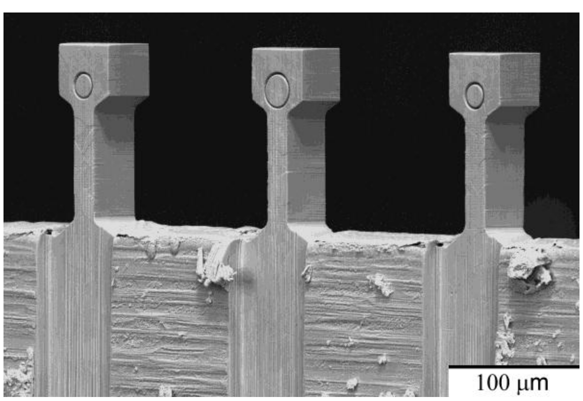

Fig. 1: Array of micro-tension samples prior to in-situ testing.

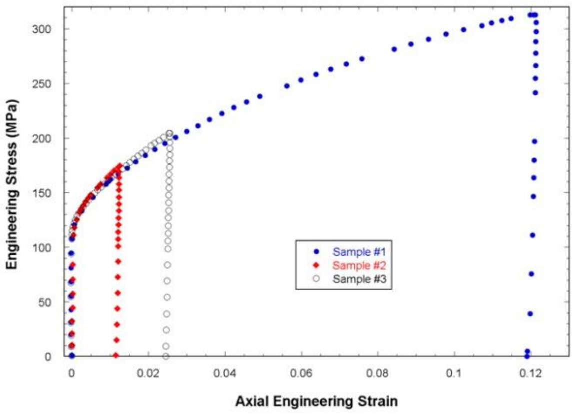

Fig. 2: Stress-strain curves for three in-situ SEM micro-tension experiments.

Fig. 3: 3D microstructure of one of the multi-crystal samples (deformed to 2.5% strain).

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